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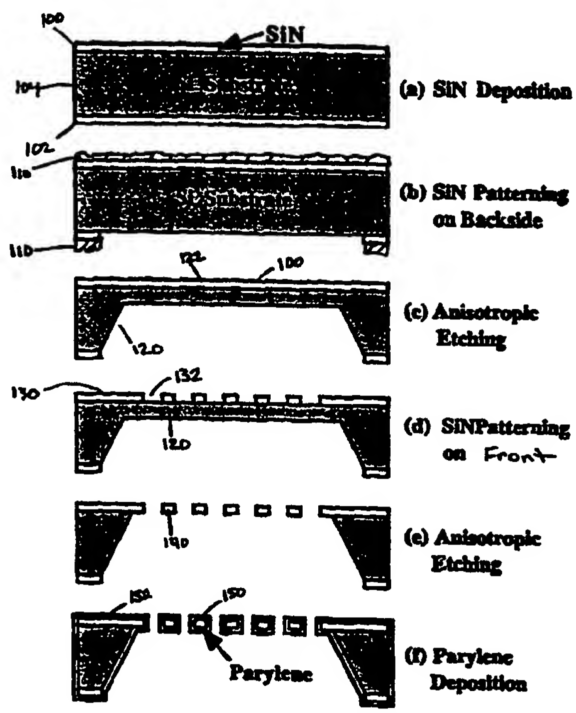
(54) Title: MICROMACHINED MEMBRANE PARTICLE FILTER USING PARYLENE REINFORCEMENT

(57) Abstract

A micromachined membrane particle filter is formed by etching a silicon substrate (104) and silicon nitride overlayers (100 and 102) and coating the entire membrane structure with a layer of parylene (150) for the purposes of imparting the characteristics of strength and biocompatibility.



Membrane Filter Fabrication Process

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MICROMACHINED MEMBRANE PARTICLE FILTER USING PARYLENE REINFORCEMENT

Statement as to Federally Sponsored Research

The U.S. Government may have certain rights in this invention pursuant to Grant No. N66001-96-C-8632 awarded by the U.S. Navy.

Background

A mechanical filter can be used to remove, filter, or collect particles. This filtering and collection of particles can be used for sampling of particles, chemical detection, and/or biological cell analysis.

It is known to make such filters using micromachining techniques to form small features in a silicon wafer. For example, this has been described in C.J.M. Van Rijin and M.C. Elwenspoek, "Micro Filtration Membrane Sieve with Silicon Micro Machining for Industrial and Biomedical Applications," Proceedings of IEEE Micro Electro Mechanical Systems Workshop (MEMS'95), pp. 83-87, 1995 and G. Kittilsland and G. Stemme, "A Submicron Particle Filter in Silicon," Sensors and Actuators, A: Physical, Vol. 23, pp. 904-907, 1990. However, the present disclosure describes a different way of developing filters which has certain improved characteristics.

One problem with prior micromachined filters is their overall strength.

Summary

A membrane particle filter is described which uses micromachining technologies. The filters are fabricated using a substrate membrane that is perforated with holes. The holes can have different shapes, different dimensions, and different opening factors. Preferred shapes include circular, hexagonal, and rectangular, with dimensions ranging from 6-13 µm.

In a preferred mode, a layer of Parylene material is uniformly coated on the filters and on the inner surfaces of the holes in order to increase the overall strength of the filter.

Another important feature is the amount of power which is necessary to provide the desired pressure drop across the filter. Proper control of the opening size allows determining various tradeoffs, including the energy and power necessary to form the desired pressure drop. Another feature of this disclosure is that the sizes of the openings can be more specifically controlled by deposition of Parylene material.

Brief Description of the Drawings

These and other aspects will now be described in detail with reference to the accompanying drawings, wherein:

FIGs. 1A-1F show a membrane filter fabrication process;

FIG. 2 shows different results of different filtering regions;

FIGs. 3A-3D show various fabricated membrane filters and their openings; and

FIGs. 4 and 5 show different pressure drops as functions of the flow rate.

Description of the Preferred Embodiments

The fabrication process for the preferred mode filter is shown in FIGs. 1A-1F. The process begins in FIG. 1A by depositing a layer of low stress LPCVD silicon nitride 100 and 102 ("SiN") at 850°C on a silicon substrate 104. The layer 100 is preferably between a ½ and 1μm micron thick. The material is deposited with an $SIH_2Cl_2 : NH_3$ gas flow ratio of 4 to 1. The SiN layers 100, 102 are deposited on both sides of silicon substrate 104.

FIG. 1B shows patterning the SiN layer 102 on the backside of the silicon substrate 104. First, the desired areas to be protected are covered with photoresist layer 110. This is followed by dry etching the SiN 102 to form a pattern.

FIG. 1C shows the anisotropic etching step which uses an anisotropic etchant such as KOH. This leaves a window 120 left in the wafer, with only a thin area of the silicon wafer 122 remaining. The thin wafer area can be between 20 and 100 μm thick. The overlying silicon nitride 100 remains unchanged during this step.

FIG. 1D shows using RIE to provide patterns 130 in the SiN layer 100. The pattern includes holes 132 into the silicon nitride layer 100. The holes can be of any desired size and shape as described above.

This is followed by placing the wafer into another anisotropic solution (for example, KOH) to remove the remaining silicon layer 120. This frees the membrane to form the unsupported membrane shown in FIG. 1E. The membrane includes unsupported silicon nitride portion 140, defining holes therebetween. These holes, however, would have low structural integrity.

Finally, a layer of Parylene is deposited over the entire wafer. Parylene is available from Specialty Coating Systems, Inc., 5707 West Minnesota Street, Indianapolis, IN 46241. The Parylene uniformly covers the entire wafer surface, forming Parylene overlayers 150, 152. Each of the holes therefore includes a Parylene overlayer on each of its surfaces: top surface, bottom surface, and all sides.

The layer of Parylene 150 serves three main purposes. The Parylene overlayer greatly improves the strength of the membrane filter by providing a reinforcement to the filter. Uniform deposition of Parylene also allows changing the hole size. Different hole sizes can be obtained from the same basic filter skeleton. Control of the thickness of the Parylene layer can be used to obtain these different hole sizes. For example, a 10 µm opening can be changed to a 4 µm opening by depositing 2 µm of Parylene on the entire device, forming two, 2-µm barriers at two ends of the hole. Hence, the same basic filter can have different holes sizes by changing the thickness of the Parylene layer.

Parylene is biocompatible, making the filter suitable for biological applications.

This fabrication process can be used for various membrane filters. The preferred hole shapes include circular, hexagonal, and/or rectangular. Filters as large as 8 X 8 square millimeters can be fabricated. The opening area ratio increases as the hole size increases. The hole size also defines the filtering threshold -- the minimum size of the particles that can be blocked by the filter.

For example, a filter with a 10.6 µm diameter hole has an opening area ratio of approximately 12½%.

Hexagonal holes can provide higher opening area ratios, but cause higher stress concentration in the membrane. This effectively reduces the strength of the filter. Rectangular holes can provide a large range of opening area ratio without changing the filtering threshold. One dimension of the rectangular holes must be kept constant.

FIG. 3A shows a top view of a circular hole. FIG. 3B shows the rectangular hole, while FIGs. 3C and 3D respectively show the hexagonal holes for these filters.

FIG. 2 shows different characteristics for the filters. The far left side of the figure shows a top view of 8 millimeter X 8 millimeter area. This is defined into different non-filtering regions and filtering regions.

TABLE 1 shows how the final Parylene coating layer increases the strength of the filters. Burst pressure of various membrane filters was tested using differential pneumatic pressure across the filter membrane. The first pressure of filter 6 in TABLE 1 was more than 4 times higher when coated with 2.69 microns of Parylene.

Table 1 Filter Strength

(Filter #6, β = 43.6%)

Parylene Thickness (μm)	Burst Pressure (PSI)
0	0.9
1.38	1.9
2.69	4.2

The fluid dynamic performance of the membrane filters was also tested, and the results are shown in FIGs. 4 and 5. Each of the pressure drops is a function of flow rate per unit area was fitted with a second order polynomial function. The power to sustain a desired flow

rate is calculated by multiplying the pressure drop by the volumetric flow rate. Hence, these features show how the pressure drop across the microfilter and power requirement increase dramatically as the opening factor increases.

Although only a few embodiments have been disclosed in detail above, many modifications are possible in the preferred embodiment without undue experimentation.

What is claimed is:

1. A micromachined membrane filter, comprising:

a silicon substrate, having a plurality of holes therein; and

a Parylene coating, covering the silicon substrate, and covering all inner surfaces of all of said holes, said holes being formed with a desired size and pitch to form a desired filtering characteristic.

2. A method of forming a micromachined particle filter, comprising:

obtaining a silicon substrate;

forming a silicon derivative overlayers on the silicon substrate;

etching one side of the silicon substrate to form a thin portion of the silicon substrate;

etching the overlayer over the other side of the silicon substrate to form holes in the overlayer;

further etching the first side to totally remove silicon from the substrate below the holes; and

depositing Parylene over remaining portions of the substrate, including inner surfaces of said holes, to form a Parylene coated membrane filter.

3. A method of forming a micromachined particle filter, comprising:

forming a plurality of holes in a substrate to form a substrate with holes of a first size;

coating edges of the holes of said first size with an overlayer material of a first thickness to form second size holes smaller than said first size holes;

coating edges of the holes of said first size with an overlayer material of a second thickens to form third size holes, different than said second size.

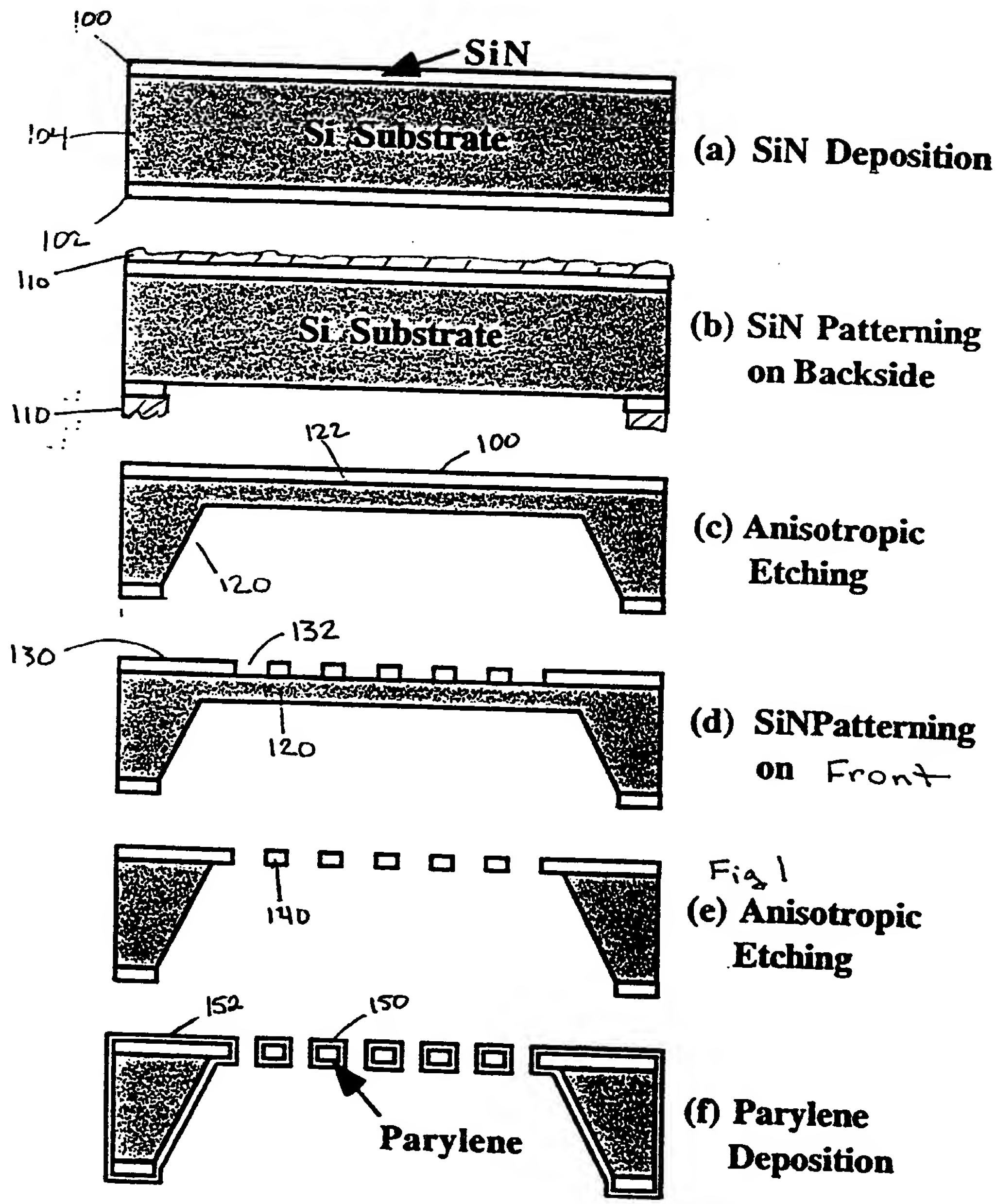


Figure 1. Membrane Filter Fabrication Process

Table 1

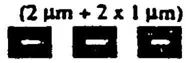
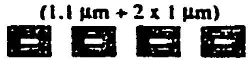
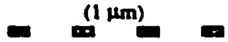
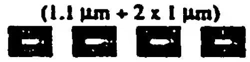
top view (8 mm x 8 mm) : non-filtering region : filtering region	cross-sectional view (thickness) : silicon nitride : parylene coating	hole size d (µm) or a x b (µm²)	opening factor β (%)
 5.3 mm	(2 µm + 2 x 1 µm) 	6	3.65
 2.3 mm	(2 µm) 	8 ~ 8.8	7.5
 5.3 mm	(2 µm) 	10.6	12.5
	(1.1 µm + 2 x 1 µm) 	7 ~ 7.5	15.1 ~ 17.4
	(1 µm) 	11 ~ 12	37.3 ~ 44.4
	(1 µm) 	6 x 20	43.6
	(1.1 µm + 2 x 1 µm) 	2~3 x 16~17	11.6 ~ 18.5

Fig. 2

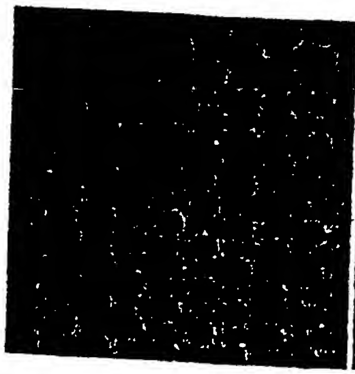


Fig 3 (a) Circular

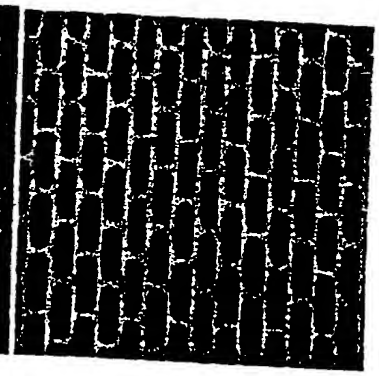


Fig3(b) Rectangular

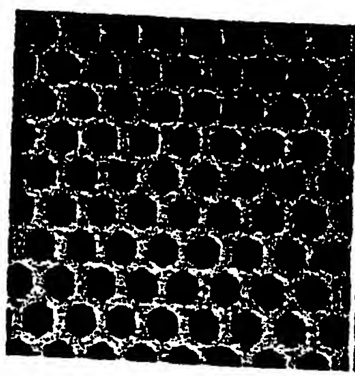


Fig 3(c) Hexagonal

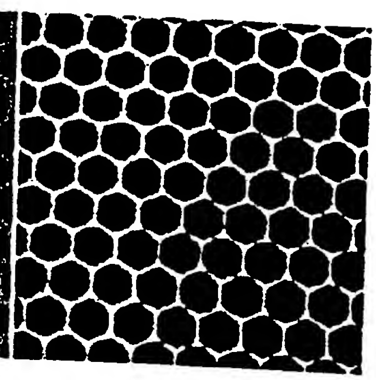


Fig3(d) Hexagonal

Figure2. Fabricated Membrane Filters

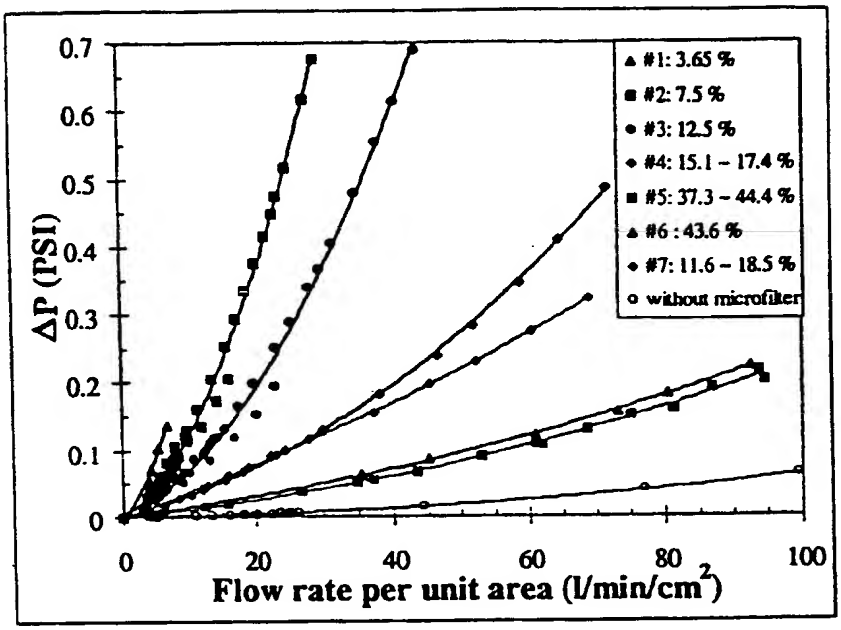


Figure 4 Pressure drop as a function of the flow rate

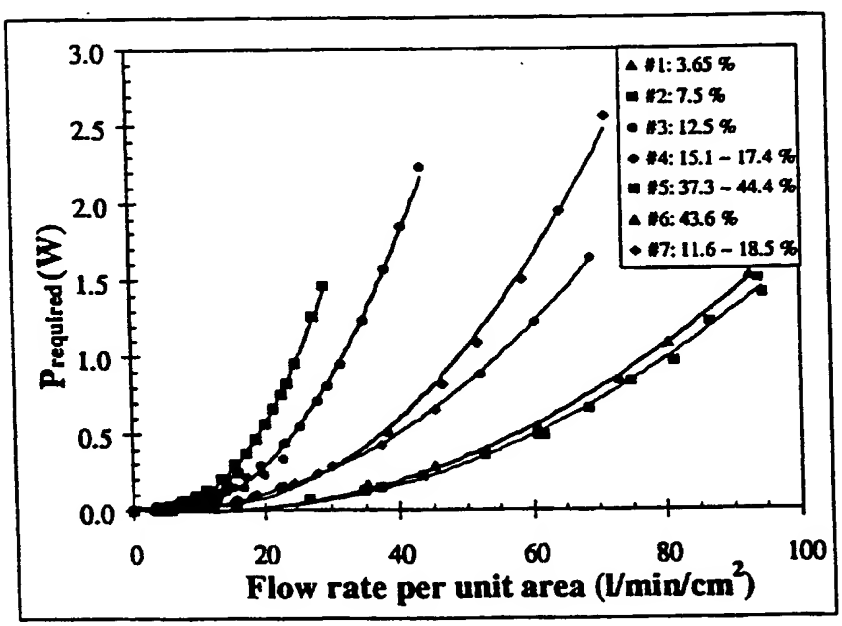


Figure 5. P wer as a functi n of the flow rate

INTERNATIONAL SEARCH REPORT	International application No. PCT/US98/23822

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :B01D 24/00; B31D 03/00
US CL : 210/506, 500.26, 500.23, 490; 216/56, 83, 95, 96, 99
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 210/506, 500.26, 500.23, 490; 216/56, 83, 95, 96, 99

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

APS
search terms: membranes, filters, biocompatible, parylene, coating (and variants thereof)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X — Y	WO 95/13860 A1 (VAN RIJN et al) 26 May 1995 (26.05.95), see entire document, especially page 8, lines 27-30, pages 17-19 and figures 31-34.	3 — 1-2
Y	US 5,067,491 A (TAYLOR, II et al) 26 November 1991 (26.11.91), see especially column 1, lines 54-59, columns 4-5, and claim 1.	1-2
A	US 5,609,629 A (FEARNOT et al) 11 March 1997 (11.03.97), see entire document.	1-2
A, P	US 5,744,360 (HU et al) 28 April 1998 (28.04.98), see especially column 11, lines 32-57.	1-3

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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